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Flaw-insensitive ceramics

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ABSTRACT

Ceramics are notorious for their 'brittleness', i.e. the sensitivity of mechanical strength to flaws on the microstructural scale. The associated notion of the 'critical flaw' has dominated design considerations concerning structural reliability and materials processing of ceramic components. This accounts for the trend over the last decade towards a processing strategy of elimination strength-degrading flaws at source. Here we propose a fundamentally different approach, that of processing ceramics with crack-impeding elements integrated into the indigenous microstructures, such that any pre-existing or service-induced flaws are effectively stabilized. Strength data on a tailored alumina/aluminium-titanate material demonstrate the capacity of our approach to produce simple ceramics with unique flaw insensitivity.

Our strategy for flaw insensitivity springs from recent studies on the strength properties of monophase aluminas and other common ceramics, using indentations to introduce flaws of controlled initial size (Cook, Lawn and Fairbanks 1985, Cook, Fairbanks, Lawn and Mai 1987, Bennison and Lawn 1989; Chantikul, Bennison and Lawn 1990). Figure 1 reproduces data for strength as a function of indentation load or corresponding indentation flaw size for aluminas of two extreme grain sizes, 2·5 and 80 μm (Chantikul, Bennison and Lawn 1990). Observe that the coarser material has a markedly reduced sensitivity to indentation load, i.e. has a greater 'flaw tolerance', albeit at the cost of a considerable loss in strength in the small flaw domain. This flaw tolerance correlates with a systematic increase in toughness as the crack extends from microstructural to macroscopic dimensions, widely known as crack-resistance, or R-curve, behaviour (Hübner and Jillek 1977, Mai and Lawn 1986, Bennison and Lawn 1989). (We emphasize that the data points in fig. 1 represent breaks exclusively from indentation flaws, so the plateau at low load reflects the intrinsic toughness characteristics and not a 'cutoff' from natural flaws.) In the case of alumina-based ceramics the R-curve is due to bridging behind the advancing tip by interlocking grains in persistent, frictional sliding contact (Knehans and Steinbrech 1982, Steinbrech, Knehans and Schaarwächter 1983, Swanson, Fairbanks, Lawn, Mai and Hockey 1987, Swanson 1988, Steinbrech and Schmenkel 1988; Vekinis, Ashby and Beaumont 1990,

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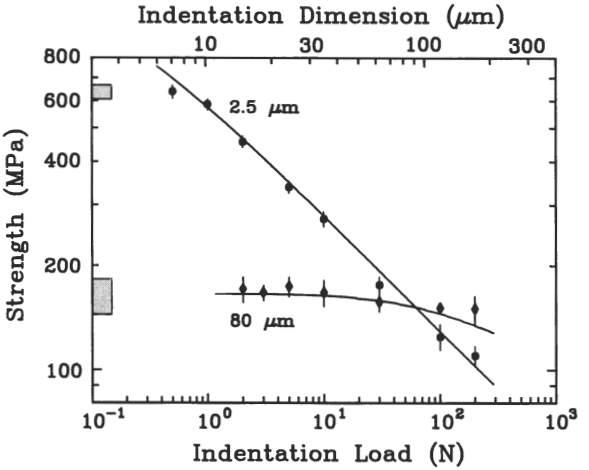
Fig. 1



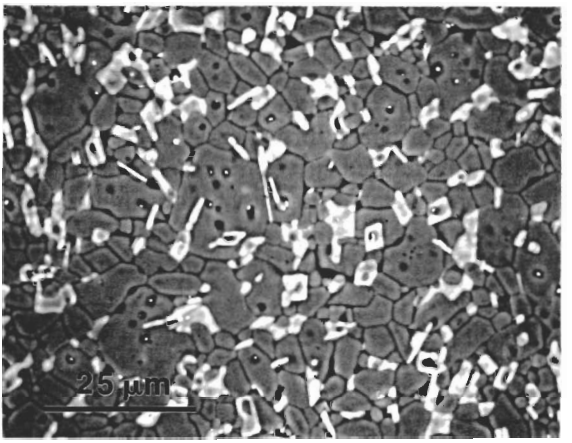
Plot of strength versus indentation load or indentation size (indentation diagonal from hardness impression) for alumina at two extreme grain sizes. Data points (from Chantikul, Bennison and Lawn 1990) represent breaks from indentation flaw sites, and indicate standard deviation limits. Solid curves are theoretical fracture mechanics fits to bridging R-curve theory (Chantikul, Bennison and Lawn 1990). Shaded areas at left represent breaks for specimens without indentation flaws.

Rödel, Kelly and Lawn 1990). There is a loose analogy here to the toughening mechanism in fibre-reinforced composites. Crack extension is impeded such that microstructural flaws, *regardless of initial size*, undergo stable extension to a critical length prior to failure. In fig. 1 the solid curves are data fits from a theoretical fracture mechanics analysis of the bridging R-curve (Chantikul, Bennison and Lawn 1990).

The above results suggest that flaw insensitivity in ceramics may be achieved by judiciously tailoring the microstructure. However, there is a limit to the benefits that may be obtained with monophase materials. Increasing the grain size scales up the crack-opening displacements over which the frictional tractions remain intact, enhancing the R-curve. It also scales up the bridge spacing, enlarging the extent flaws may grow before bridges are activated. The net result is a tradeoff: the curves in fig. 1 cross each other. An alternative, more radical route suggested by bridging theory (Bennison and Lawn 1989) is to build strong internal compressive stresses into the microstructure so as to enhance grain–grain contacts during slide-out, thereby augmenting frictional restraining forces. This brings us to our hypothesis for microstructural design of fine-grained, flaw-tolerant ceramics: *incorporate heterogeneities into the matrix using a second phase with large thermal expansion mismatch.*

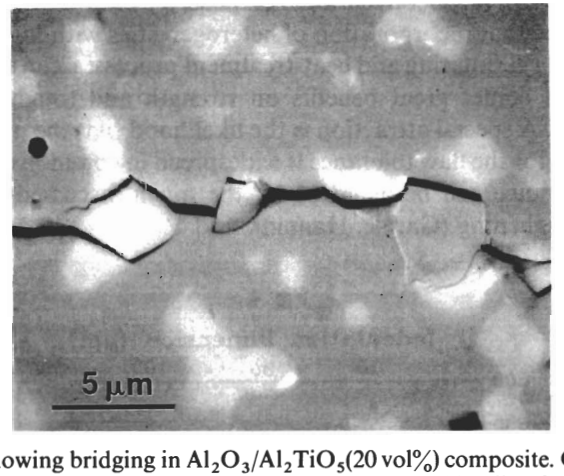
The notion of manipulating microstructures for high toughness through additive phases is not new (Claussen 1990). What is new is the specific intent of tailoring for flaw tolerance. Here we demonstrate the hypothesis with some results on an alumina matrix containing 20 vol% aluminium titanate as a second phase. Aluminium titanate was chosen because its thermal expansion coefficients show extreme anisotropy, and differ dramatically from those of alumina: Al_2O_3 (hexagonal), $\alpha_a = 9 \times 10^{-6}\,°C^{-1}$, $\alpha_c = 10 \times 10^{-6}\,°C^{-1}$; Al_2TiO_5 (orthorhombic), $\alpha_a = 12 \times 10^{-6}\,°C^{-1}$, $\alpha_b = 20 \times 10^{-6}\,°C^{-1}$, $\alpha_c = -3 \times 10^{-6}\,°C^{-1}$ (Bayer 1971). Fabrication was by a conventional pressureless sintering route (Runyan and Bennison 1991). A colloidal suspension of high-purity powders (α-Al_2O_3, Sumitomo AKP-HP grade, 99·995% pure, 0·5 μm

Fig. 2



Scanning electron micrograph of Al_2O_3/Al_2TiO_5(20 vol%) composite. Polished section, surface thermally etched at 1490°C for 6 min in air to reveal grain structure. Backscattered electron imaging: white phase Al_2TiO_5; grey phase Al_2O_3; black phase porosity.

Fig. 3



SEM micrograph showing bridging in Al_2O_3/Al_2TiO_5(20 vol%) composite. Observations made during stressing of indentation crack using *in situ* flexural loading fixture. Backscattered electron image, revealing phase structure. Second phase attracts and deflects the primary crack, thereby establishing strong friction points at grain facets for effective bridging. As the crack advances, more bridges are activated, thus increasing the toughness and stabilizing the crack.

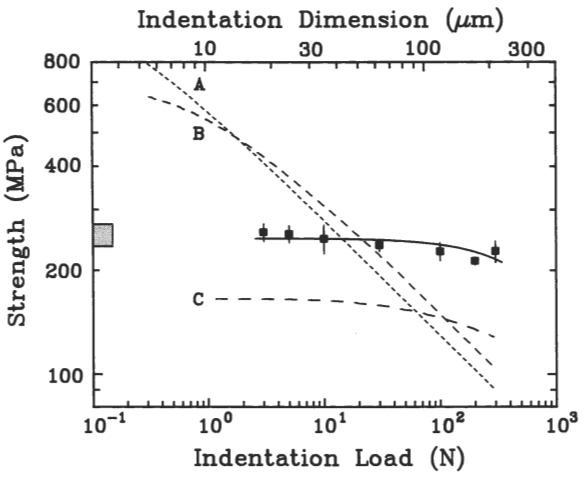
crystallites; β-Al_2TiO_5, Trans-Tech, 99·9% pure, 1–5 μm crystallites) in water (pH ~ 3) was dried, and formed into green discs by single-end die pressing (63 MPa) followed by isostatic pressing (350 MPa). The discs were calcined in air at 1050°C for 12 h and sintered at 1600°C for 1 h, to produce high-density (~99% theoretical limit) specimens with a matrix grain size of 6 μm. Figure 2 shows the microstructure. The material has an equiaxed structure with the aluminium titanate particles distributed mainly at the matrix grain boundary junctions, with occasional agglomerates of 5–10 particles. Scanning electron microscopy examination of extended indentation cracks during

actual loading to failure (Braun, Bennison and Lawn 1991) revealed evidence for copious grain-interlock bridging at crack interfaces. The example shown in fig. 3 indicates that the aluminium titanate particles play a highly active role in the bridging process.

Figure 4 plots strength versus indentation load and indentation flaw size for our 'composite' material. The solid curve through the data set is an empirical fit. For comparison, we include as the dashed curves the responses for the two homogeneous aluminas in fig. 1, plus the interpolated response for an alumina of the same base grain size as the composite (6 μm) using an analysis from an earlier study (Chantikul, Bennison and Lawn 1990). The strengths of the composite are significantly higher than for the monophase aluminas in the large-flaw size domain, although this is offset somewhat by lower strengths in the small-flaw size domain. Again, the trade-off renders the strength of the composite almost completely independent of the flaw size. Note especially that the strength level is well in excess of that for the coarsest alumina, attesting to the increased efficacy of the bridging.

Flaw insensitivity is a highly desirable property in structural ceramics from the standpoint of component design. Primarily, it enables an engineer to design to a single, well-defined stress level, without regard to the size of a critical flaw. It also provides inbuilt protection for the component against in-service damage (e.g. from impacts with errant particles in the atmosphere), and does not place undue onus on the ceramics manufacturer to resort to unconventional, expensive, ultra-clean processing routes. The implications are that incorporation of heterogeneities with high internal stresses, properly controlled via sintering and heat-treatment processes, can suitably modify the microstructure and confer great benefits on strength and toughness properties of structural ceramics. A special attraction is the likelihood that the underlying bridging process responsible for the flaw tolerance is widespread in common ceramics (Swanson 1988); similar tolerance can be achieved by the much discussed process of phase-transformation toughening (Garvie, Hannink and Pascoe 1975, Marshall 1986), but

Fig. 4



Plot of strength versus indentation load for alumina/aluminium-titanate composite (data points, solid curve). Shaded area at left represents breaks for specimens without indentation flaws. Dashed curves for alumina of different grain sizes included for comparison: (A) 2·5 μm and (C) 80 μm, from fits in fig. 1; (B) 6 μm, theoretical prediction.

that mechanism has thus far been demonstrated in only one material, zirconia. Again, we would stress that our strategy is counterintuitive to the more traditional procedures that seek to remove all flaws by progressively refining the microstructure (Lange 1983; Lange and Metcalf 1983, Lange, David and Aksay 1983, Alford, Birchall and Kendall 1987, Kendall, Alford, Clegg and Birchall 1989). Those procedures certainly produce materials of higher laboratory strength: but the processing is exacting and costly; the fine scale of the defect structure does not lend itself to nondestructive evaluation; and, most important, the materials are highly susceptible to subsequent in-service strength degradation.

The future challenge is to tailor heterogeneous ceramics which preserve the quality of flaw insensitivity displayed here and at the same time attain even higher strength levels.

Acknowledgments

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